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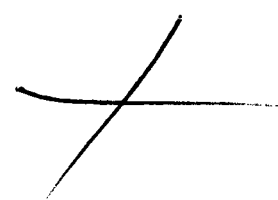
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# DEVELOPMENT OF DISPERSION STRENGTHENED TANTALUM BASE ALLOY

Fourth Quarterly Report

by

R. W. Buckman, Jr.

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
LEWIS RESEARCH CENTER  
UNDER CONTRACT NAS3-2542

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DEVELOPMENT OF DISPERSION STRENGTHENED  
TANTALUM BASE ALLOY

by

R. W. Buckman, Jr.

FOURTH QUARTERLY PROGRESS REPORT

Covering the Period

August 20, 1964 - November 19, 1964

Prepared For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
Contract NAS 3-2542

Technical Management  
Paul E. Moorhead  
NASA - Lewis Research Center

Astronuclear Laboratory  
Westinghouse Electric Corporation  
Pittsburgh 36, Pa.

## FOREWORD

This report was prepared by the Astronuclear Laboratory of the Westinghouse Electric Corporation under Contract NAS 3-2542. This work is administered under the direction of the Nuclear Power Technology Branch of the National Aeronautics and Space Administration with Mr. P. E. Moorhead acting as Technical Manager.

This work is being administered at the Astronuclear Laboratory by R. T. Begley, with R. W. Buckman, Jr. serving as principal investigator. This report covers the work performed during the period August 20, 1964 to November 19, 1964. Other Westinghouse personnel contributing are:

A. Filippi	Melting and Primary Breakdown
G. G. Lessmann	TIG Welding
D. R. Stoner	EB Welding
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J. Salatka*	

\* Westinghouse Research and Development Center



## ABSTRACT

1-7199

Development of dispersion strengthened tantalum base alloys for use in advanced space power systems continued with the tentative selection of three compositions which will be melted as 4 inch diameter ingots. Continuing investigations of response to heat treatment has shown that Ta-W-Zr-N and Ta-W-Mo-Re-Zr-N alloys are age hardenable systems which show promise for use as turbine materials. A procedure for the addition of nitrogen to first melt electrodes has been established.

*Author*

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Westinghouse Electric Corporation

Astronuclear Laboratory

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February 16, 1965

SUBJECT: Contract NAS 3-2542

Dear Sir:

As directed by the National Aeronautics and Space Administration, enclosed is a copy of the 4th quarterly report, "Development of Dispersion Strengthened Tantalum Base Alloy", describing the activities of this laboratory under subject contract during the period August 20, 1964 thru November 19, 1964.

Any comments or suggestions concerning this report are welcomed and should be directed to the undersigned.

Very truly yours,

R. T. Begley  
Supervisory Engineer  
Materials Department

R. W. Buckman, Jr.  
Materials Department

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## I. INTRODUCTION

This is the fourth quarterly progress report on the NASA supported program "Development of a Dispersion Strengthened Tantalum Base Alloy". This report describes the work conducted during the period August 20, 1964 to November 19, 1964. This program is a continuation of the tantalum alloy investigation being conducted under Contract NAS 3-2542.

The previous phase of this program was concerned with the development of a dispersion strengthened tantalum base alloy for use in the 2400 to 3000°F operating temperature range of advanced space power systems. For this application resistance to creep deformation is the primary strength consideration. However, since the component hardware is to be fabricated from sheet and/or tubing, weldability and fabricability are also important criteria.

During Phase I of this work, significant improvements in creep resistance of tantalum were achieved with minimum degradation of fabricability and weldability. Also, tantalum alloy compositions which showed promise for turbine applications were developed. Some of the more cogent findings of Phase I are outlined below and include:

1. Tantalum with approximately 9w/o W, 1w/o Hf and with carbon additions up to 0.03w/o gave fabricable and weldable tantalum alloys with superior creep resistance at 1315°C (2400°F) over any commercially available tantalum base alloy.
2. Substitution of 1.5w/o Re for part of the tungsten and/or replacement of carbon with nitrogen resulted in still further improvement in creep resistance without seriously affecting fabricability and weldability. Creep data for promising compositions are listed in Table I.
3. Optimum resistance to creep deformation was obtained when the interstitial (C+N) to reactive metal (Hf+Zr) atom ratio was maintained at stoichiometry.
4. The creep behavior of tantalum base alloys containing reactive metal (Hf or Zr) additions was strongly dependent on the test pressure, and testing at pressures of  $\leq 10^{-8}$  torr was necessary to prevent contamination of the test specimen. It was demonstrated experimentally that contamination of T-111 (Ta-8W-2Hf) during creep testing at  $10^{-6}$  torr in an oil diffusion pumped system resulted in a significant increase in creep resistance due to the formation of finely dispersed oxides and carbides of hafnium.

The primary objective of Phase II is the scale-up of three compositions to 4 inch diameter ingots. Two compositions will be selected for sheet and tubing applications, and one composition will be selected for use as turbine material.

TABLE I - Creep Properties of Developmental Tantalum Base  
Alloys at 2400°F and  $10^{-8}$  Torr

Composition (Heat No.)	Stress (psi)	Test Time (Hrs.)	Total Elongation (%)	$\dot{\epsilon}$ Min. % Per Hr.	Time to 1% Strain (Hrs.)
Ta-8W-2Hf(T-111)	14,100	73	2.56	0.014	41
Ta-9.6W-2.4Hf-0.01C (T-222)	14,500	170	2.11	0.0082	110
Ta-9W-1Hf-0.025C (NASV-9)	15,160	300	2.0	0.0052	180
Ta-5.7W-1.56Re-.7Mo -0.25Hf-0.13Zr -0.017C-0.02N(NAS-36)	15,000	193	0.25	0.0012	770*
Ta-7.1W-1.56Re -0.26Zr-0.02N (NAS-38)	15,390	94	0.2	0.002	480*

\* Extrapolated

In addition, work from Phase I is being continued to identify the dispersed second phase(s) responsible for improving creep resistance and to determine stability, morphology, and composition of the precipitating phase. Authorization has been received from the cognizant NASA personnel to proceed with the investigation of additional alloy optimization prior to final selection of the compositions to be melted as 4 inch diameter ingots.

## II. PROGRAM STATUS

### A. PHASE II TECHNICAL PROGRAM OUTLINE

Prior to the initiation of the phase II scale-up, further optimization of the alloy compositions is being investigated. Upon completion of the alloy optimization investigation, three tantalum alloy compositions will be selected and melted as 4 inch diameter ingots. One composition will be selected for use as turbine material and two compositions will be selected for optimum resistance to creep deformation while maintaining good fabricability and weldability characteristics for use in tubing applications.

The compositions selected for tubing applications will be melted into 4 inch diameter ingots utilizing AC plus DC or double AC vacuum arc melting. The as-cast ingot will be processed to 0.040 inch thick by the schedule shown in Figure 1. The 0.040 inch thick sheet will be evaluated for weldability, fabricability, short-time tensile properties, and resistance to creep deformation as outlined in Figure 2. The importance of the testing environment on properties during creep has been demonstrated previously<sup>1</sup> and all creep tests will be conducted at pressures not exceeding  $1 \times 10^{-8}$  torr.

The composition for the turbine material will also be melted as a double vacuum arc melted ingot 4 inches in diameter by 8 inches long. The turbine composition will be processed to upset forged discs 4 inches in diameter by 1/2 inch thick by the processing schedule outlined in Figure 3. Two different processing routes will be followed to obtain upset forged discs. Since weldability is of no importance in turbine applications, strength and forgeability are the primary characteristics which are to be evaluated. Notched tensile transition temperature and creep strength are the primary strength parameters being evaluated. Evaluation of the forging composition will be done according to the outline in Figure 4. Test bars of the forging composition will be cut from the as-forged discs.

Creep tests of 1000 hour duration will be conducted at pressures of less than  $10^{-8}$  torr. Elevated temperature tensile tests will be done at pressures of less than  $10^{-5}$  torr.



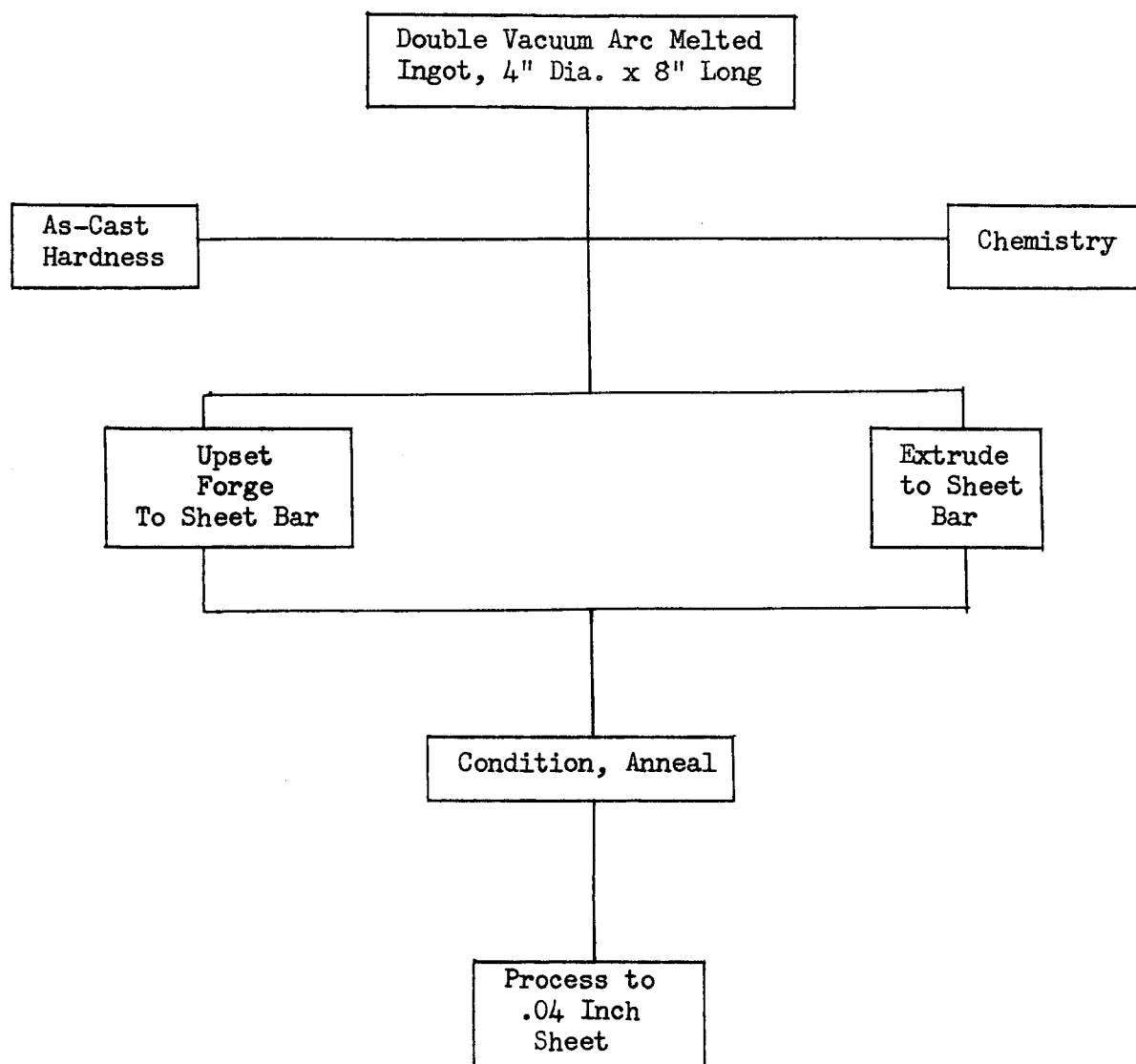


FIGURE 1 - Schedule for Sheet and Tubing Alloys

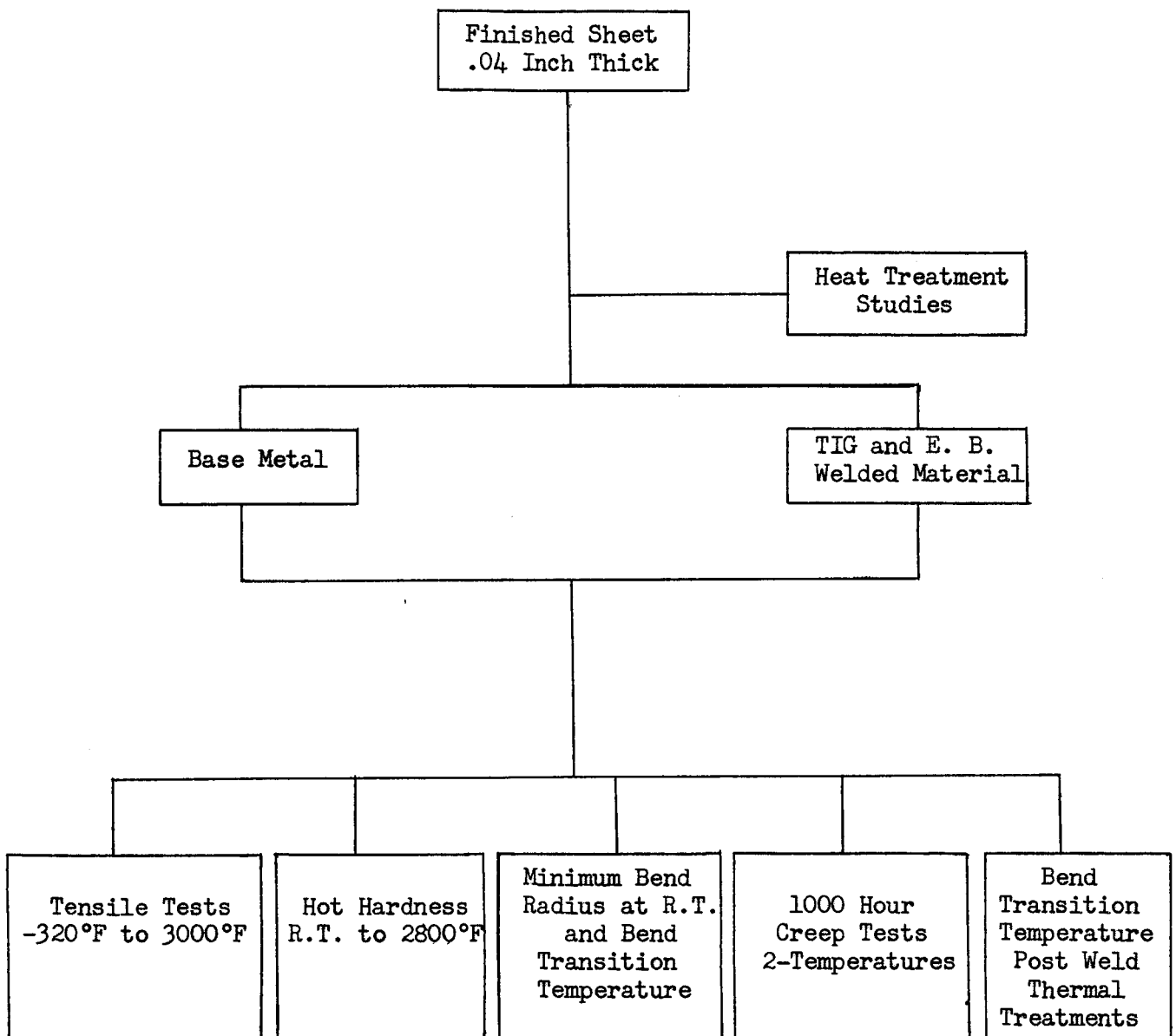


FIGURE 2 - Evaluation Schedule for Sheet and Tubing Alloys

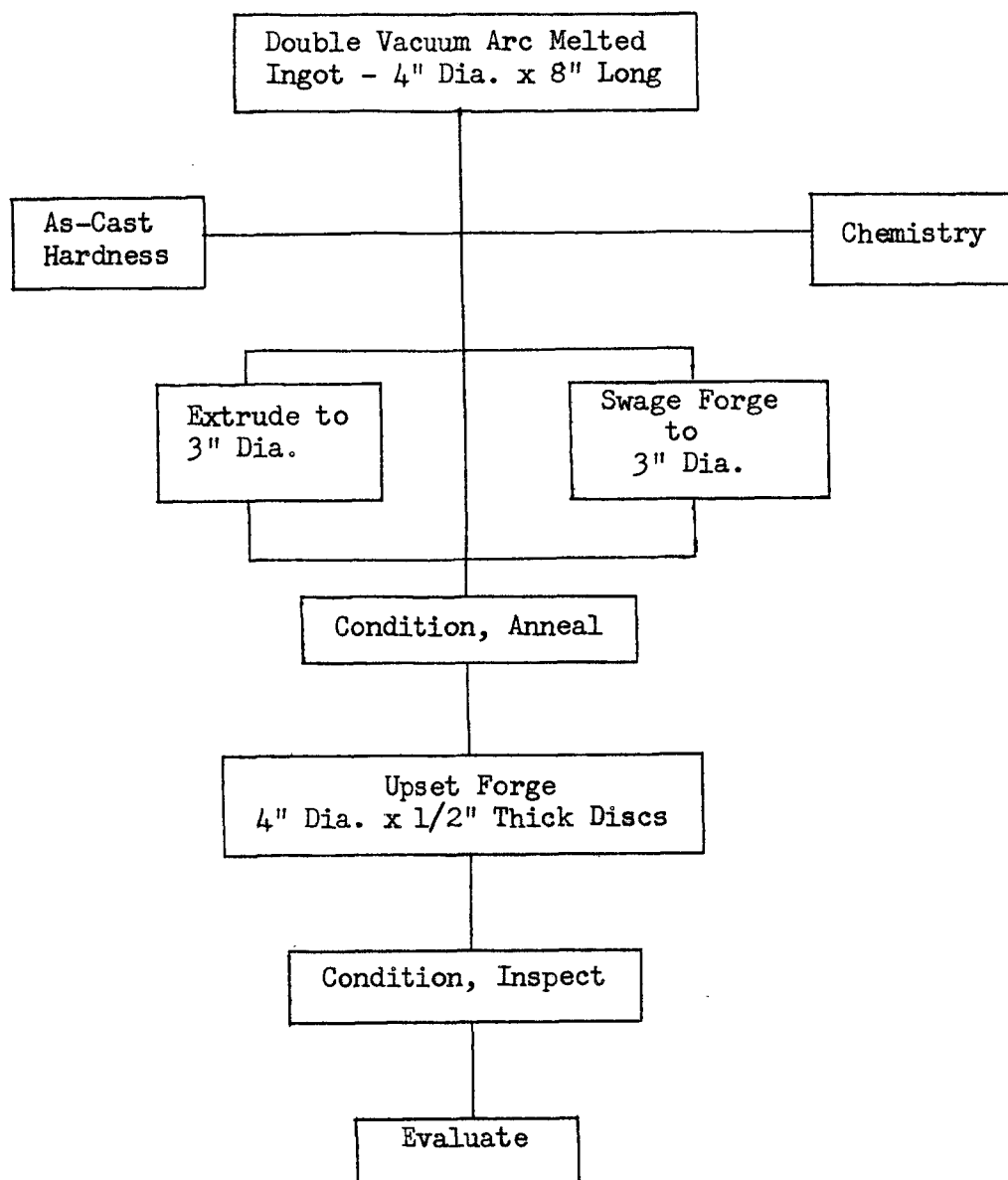


FIGURE 3 - Processing Schedule for Turbine Alloy

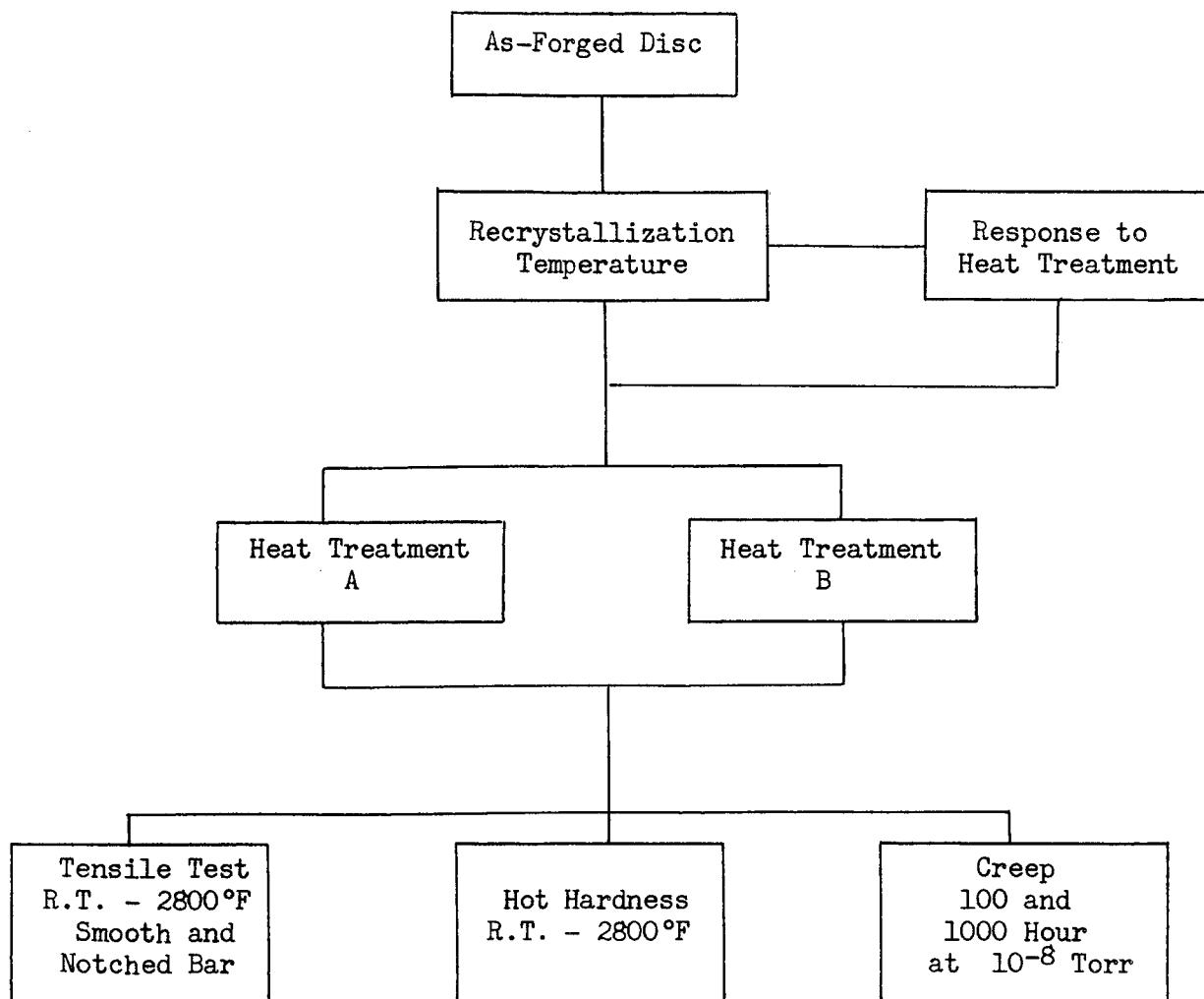


FIGURE 4 - Evaluation Schedule for Turbine Alloy

## B. MATERIALS PROCUREMENT

During this period the required starting material for all three 4 inch diameter ingots was obtained, and consisted of unalloyed tantalum, Ta-10W, and unalloyed rhenium. The vendor analyses for the Ta-10W and unalloyed Ta, which was purchased as 1/4 inch plate, are listed in Table II. Rhenium (99.99%) was purchased as 0.02 inch thick x 2 inch wide strip.

## C. ULTRA-HIGH VACUUM CREEP EQUIPMENT

An additional Varian ultra-high vacuum creep furnace was received and installed. This is the third such unit procured by Westinghouse for determining creep behavior of the developmental tantalum alloys. The system, shown in Figure 5, is equipped with a water cooled bell jar and a Weston thermal-watt converter for controlling power input to the furnace. Varian has upgraded the 400 l/s sputter ion pump, with which the other two units are equipped, to 500 l/s pumping speed by modifying the pump cell geometry.

The base pressure of the empty system at ambient temperature after a 21 hour bakeout was less than  $5 \times 10^{-10}$  torr. Maximum temperature obtained on the exterior surface of the system during bakeout was 188°C (370°F) on the upper bell jar and 116°C (240°F) on the weight sump. With a power input of 5 K.W., the furnace element was operating at 1950°C (3542°F). Temperature was measured using a Pyro micro-optical pyrometer. Heating element temperature as a function of power input is shown in Figure 6. At 1950°C (3542°F) the maximum pressure<sup>(a)</sup> was  $4 \times 10^{-8}$  torr and a pressure of  $1 \times 10^{-8}$  torr was obtained after four hours at temperature.

A titanium sublimation pump has also been added to the third creep system and pressures of  $2 \times 10^{-10}$  torr at 1700°C (3090°F) were maintained on the empty system with periodic use of the pump (60 seconds of use every 72 hours). Additional tests are underway to further evaluate the use of titanium sublimation pumping during creep testing.

## D. TWO-INCH DIAMETER INGOT MELTS

Two compositions, NASV-10 (Ta-7.1W-1.56Re-0.25Hf-0.13Zr-0.03N) and NASV-11 (Ta-9.0W-1.5Re-1Hf-0.015C-0.015N) were double vacuum arc melted as 2 inch diameter ingots. Melting data for NASV-10 and NASV-11 are given in Table III. These two compositions were selected for turbine applications, hence forgeability and strength are the prime property considerations. The NASV-10 composition was melted during Phase I as an 800 gram button ingot, heat number NAS-39. This composition had excellent creep resistance at 2400°F, i.e. an 0.21% total elongation at a stress of 20,000 psi in 197 hours.<sup>1</sup>

---

(a) Pressure based on ion pump current

TABLE II - Vendor Analysis of Starting Material

Element	Ingot Analysis, <sup>(1)</sup> ppm	
	Unalloyed Tantalum	Ta-10W
Al	< 10	---
B	< 1	---
C	< 30	< 10
Cb	245	95
Cd	< 1	---
Co	< 5	< 10
Cr	< 10	---
Cu	< 2	---
Fe	20	< 10
H	3	2
Mg	< 10	---
Mn	< 10	---
Mo	< 10	< 10
N	< 27	< 10
Ni	< 10	< 10
O	< 50	< 10
Pb	< 5	---
Si	24	---
Sn	< 10	---
Ti	< 10	---
V	< 10	---
W	275	9.94%
Zn	< 10	---
Zr	< 50	---

(1) Average of two ingot locations for unalloyed tantalum and of five locations for the Ta-10W.

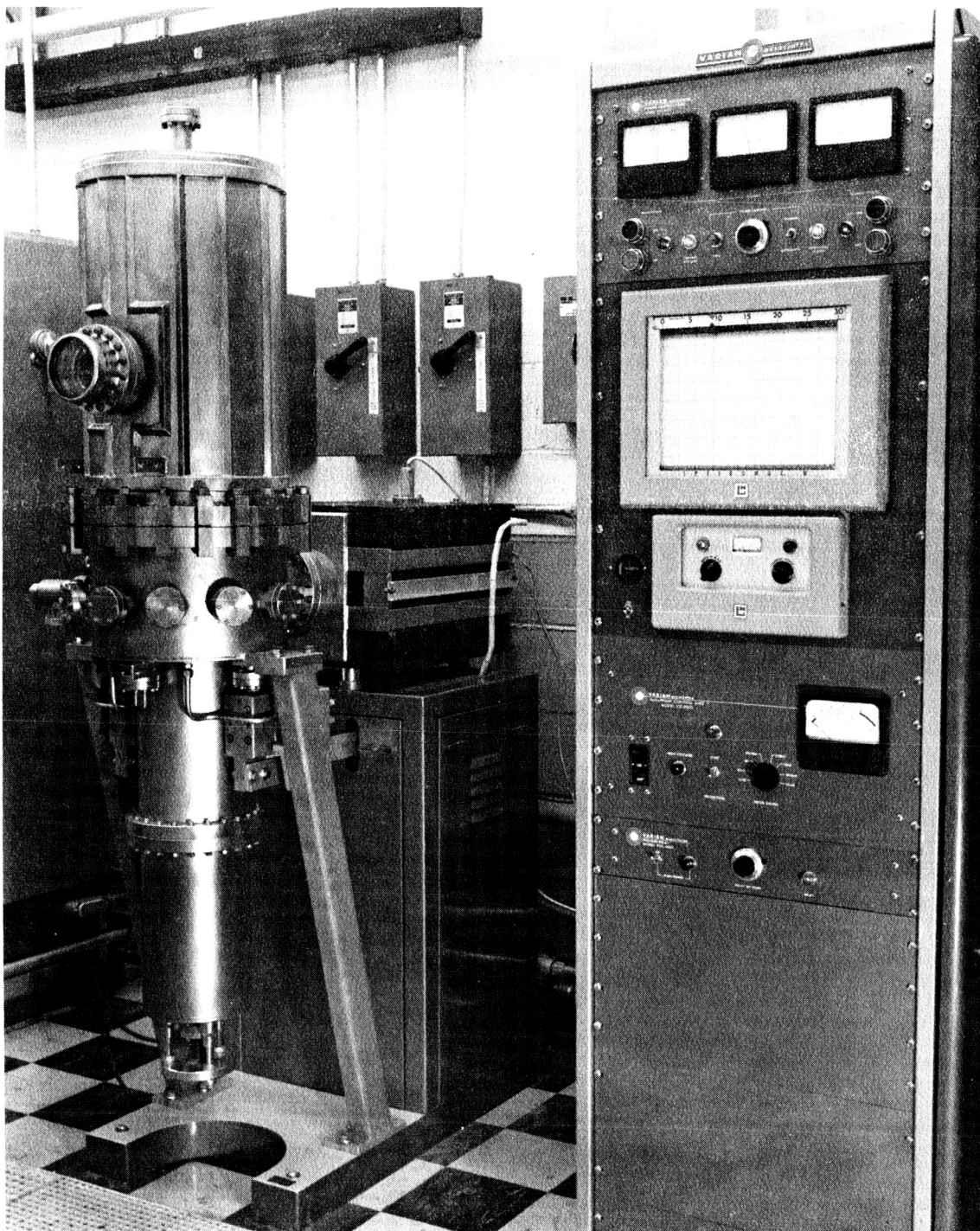


FIGURE 5 - Ultra-High Vacuum Creep System

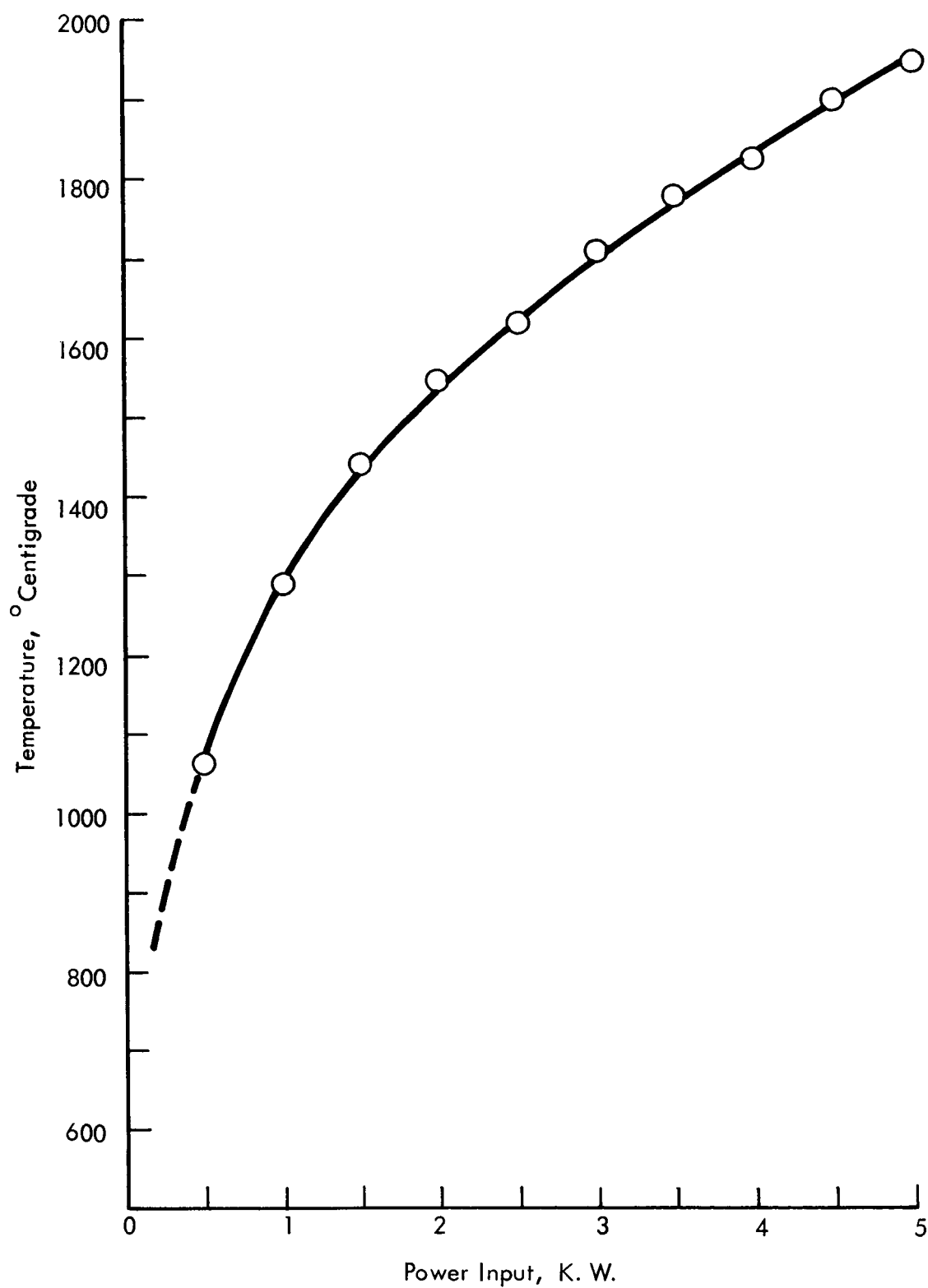


FIGURE 6 - Power Input vs. Heating Element Temperature for UHV 3



TABLE III - Melting Data

Composition (Heat No.)	Melting		Ingot Diameter (Inches)	Melt Rate (lbs/minutes)
	Current (Amps)	Volts		
Ta-7.1W-1.56Re (1st Melt)	1950	20	1.4	1.8
-0.25Hf-0.13Zr (2nd Melt)	2400	20	2.0	3.8
-0.03N (NASV-10)				
Ta-9W-1.5Re-1Hf (1st Melt)	1800	22-24	1.4	1.7
-0.015C-0.015N (2nd Melt)	2400	22	2.0	3.1
(NASV-11)				

Samples taken from each ingot were annealed for one hour at temperatures between 1200-2000°C (2190-3632°F). The microstructure of NASV-10 was single phase as-cast and after the various one hour thermal treatments. NASV-11 in the as-cast condition contained a fine precipitate decorating a sub-boundary network. There was little change in this microstructure after the one hour heat treatments. Table IV contains the room temperature hardness of the as-cast and heat treated samples. The various one hour annealing treatments had little effect on the as-cast hardness.

The as-cast ingots were lathe conditioned to 1.8 inch diameter, and cut in half to produce two extrusion billets per ingot. A molybdenum nose plug was pinned to one end of the ingot section and then the entire billet was plasma sprayed under a protective atmosphere with unalloyed molybdenum to give a coating thickness of 0.015-0.025 inches. The molybdenum coating afforded oxidation protection during heating and also provided some lubricity during extrusion. Both compositions were extruded to round bar using the Dynapak Model 1220C, a high energy rate machine. Extrusion data for all four billets is given in Table V. The as-extruded rods were hot straightened by swaging and then lathe conditioned prior to further processing. The lathe conditioned extrusion will be annealed for one hour at 1650°C (3000°F) and then swaged to 3/8 inch diameter rod at 400°C (750°F). The one hour recrystallization temperature will be determined and tensile and creep properties will be obtained on the as-swaged 3/8 inch diameter rod.

#### E. NITROGEN ADDITION TO TANTALUM ALLOYS

Additions of nitrogen in excess of 300 ppm to the first melt sandwich electrodes for consumable electrode vacuum arc melting is difficult since the electrode construction requires that the nitrogen be added as master alloy in sheet form. Thus, the present technique of preparing a fabricable tantalum-nitrogen master alloy addition limits the amount of nitrogen that can be conveniently added, to less than 300 ppm. To circumvent this limitation and also to reduce the cost of the nitrogen addition, gas nitridation of tantalum sheet was investigated. Nitrided strips of the appropriate dimensions could then be easily assembled into the first melt electrode.

Begley, et al made large nitrogen additions (~.2%) to columbium alloys in a similar manner, and a review of the literature<sup>2</sup> had indicated that nitridation of tantalum should be straightforward.

Unalloyed tantalum strips 1/2 inch wide x 15 inches long x 0.06 inch thick were the size multiple that was compatible with the experimental equipment and the first melt electrode configuration. A schematic representation of the experimental set-up is shown in Figure 7. The tantalum strips were positioned in the muffle and then the system was evacuated to  $10^{-5}$  torr and then backfilled,

TABLE IV - Room Temperature Vickers Hardness\*

Condition	Ta-7.1W-1.56Re-0.25Hf -0.13Zr-0.03N (NASV-10)	Ta-9.0W-1.5Re-0.0Hf -0.015C-0.015N (NASV-11)
As-Cast	312	361
A.C.+1 Hr. at 1200°C	298	359
A.C.+1 Hr. at 1400°C	293	342
A.C.+1 Hr. at 1600°C	303	359
A.C.+1 Hr. at 1800°C	303	348
A.C.+1 Hr. at 2000°C	295	347

\* 30 Kg Load

TABLE V - Extrusion Data

Heat No.	Billet Size		Extrusion		Extrusion	
	Length Inches	Dia. Inches	Temperature (°C)/(°F)	Ratio	Length Inches	Dia. Inches
NASV-10 B	2-3/8	1.8	1500/2732	4.8:1	8	0.86
NASV-10 T	2	1.8	1550/2822	4.8:1	10-1/2	0.86
NASV-11 T	1-9/16	1.8	1500/2732	4.2:1	6-3/8	0.92
NASV-11 B	1-9/16	1.8	1400/2552	4.2:1	6-1/2	0.90

Remarks: Extrusion billets plasma sprayed with unalloyed molybdenum.

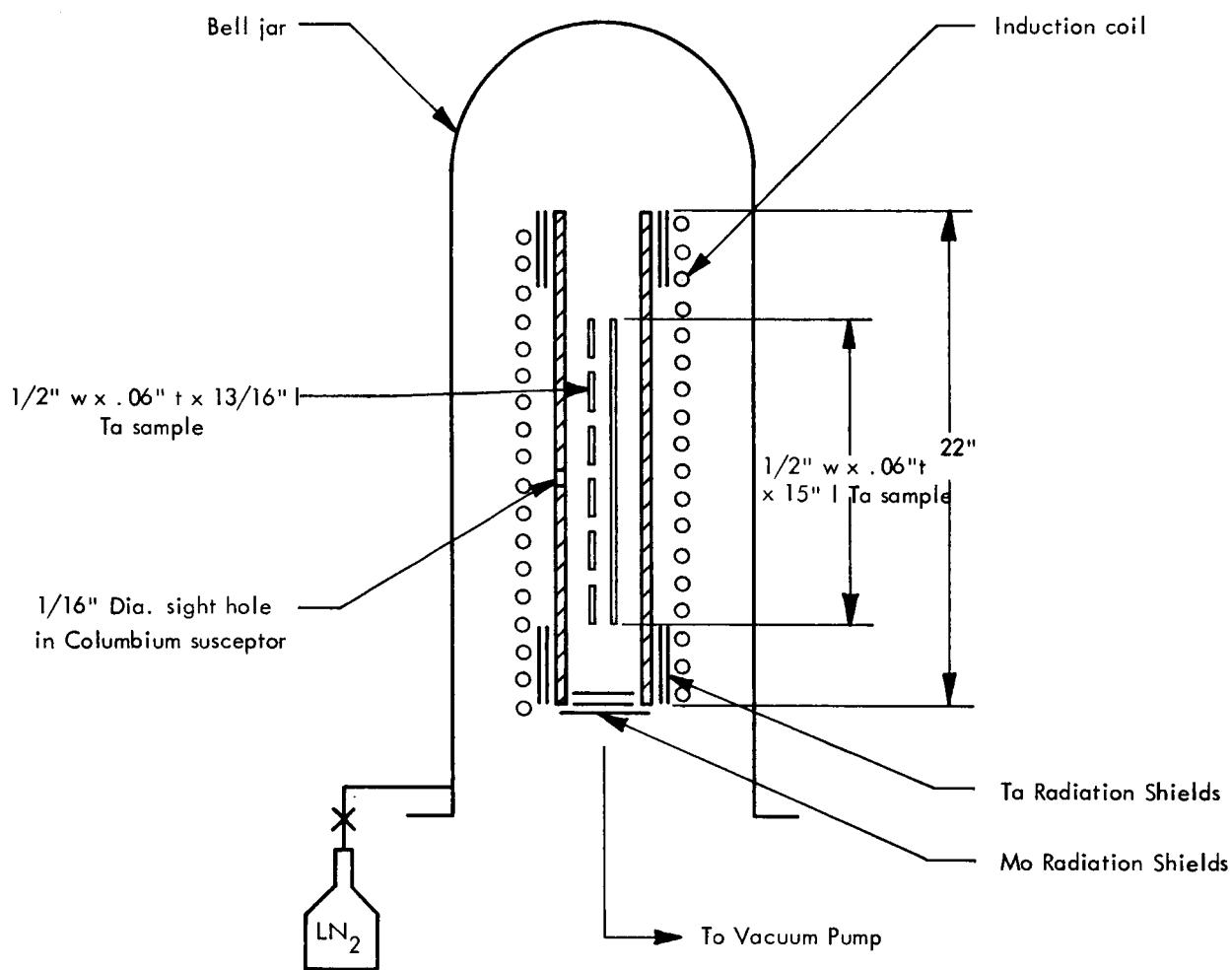


FIGURE 7 - Experimental Set-Up for Nitridation of Tantalum

by boil off from a liquid nitrogen filled dewar, to give a nitrogen partial pressure of 0.194 atmospheres. Heating was accomplished by radiation from an inductively heated columbium susceptor, 1-1/2 inch O.D. x 1-1/4 inch I.D. x 22 inches long. Temperature measurements were made with a L & N disappearing filament optical pyrometer. Test strips were carefully weighed before and after the test and nitrogen pick-up was based on weight gain. The test strips were etched in a  $\text{HNO}_3$ -HF mixture prior to nitriding.

Nitrogen pick-up as a function of temperature and time is shown in Figures 8 and 9. The time variation of the reaction at 1550°C (2820°F) fits the parabolic law. Gulbransen and Andrews<sup>3</sup> observed a similar behavior at 700°C (1290°F) and at 0.1 atmosphere of  $\text{N}_2$ . Seven specimens, 13/16 inch long x 1/2 inch wide x 0.06 inch thick were distributed over a 15 inch length to check variation in nitrogen pick-up along the length of the furnace. Variation of nitrogen concentration as a function of furnace position is shown in Figure 10.

The results indicate that nitrogen variation along the length of the strip will be within  $\pm 0.01\%$  of the desired value. Thus, based on the first melt electrode configuration, nitrogen concentration in the final ingot should be controlled to within  $\pm 10\%$  of the desired amount. For example, an addition of 100 ppm nitrogen will be within the range of 90-110 ppm.

#### F. RESPONSE TO HEAT TREATMENT

Since both carbon and nitrogen have a decreasing solubility in the tantalum alloy matrix, with decreasing temperature, thermal treatments can cause significant changes in properties. The following compositions were selected for studying changes in properties caused by various thermal treatments:

Ta-8.6W-0.53Hf-0.02C (NAS-21)  
Ta-5.7W-1.56Re-0.7Mo-0.25Hf-0.13Zr-0.015N-0.015C (NAS-36)  
Ta-7.1W-1.56Re-0.25Hf-0.13Zr-0.03N (NAS-39)  
Ta-5.3W-1.56Re-0.65Mo-0.52Zr-0.05N (NAS-42)  
Ta-6.5W-0.8Mo-0.5Hf-0.26Zr-0.017C-0.02N (NAS-44)  
Ta-8.1W-0.52Zr-0.08N (NAS-49)

All five compositions selected had a total of 9 atom percent substitutional solute (W+Mo+Re+Hf+Zr) addition and contained interstitial carbon and nitrogen additions within the range of 200-800 ppm. Sheet samples, 0.04 inch thick were heated to 2000°C (3630°F) in vacuum of  $1 \times 10^{-5}$  torr, held at temperature for one hour and then quenched by admitting helium gas to the system. The time from 2000°C (3630°F) to black heat was approximately 90 seconds. In

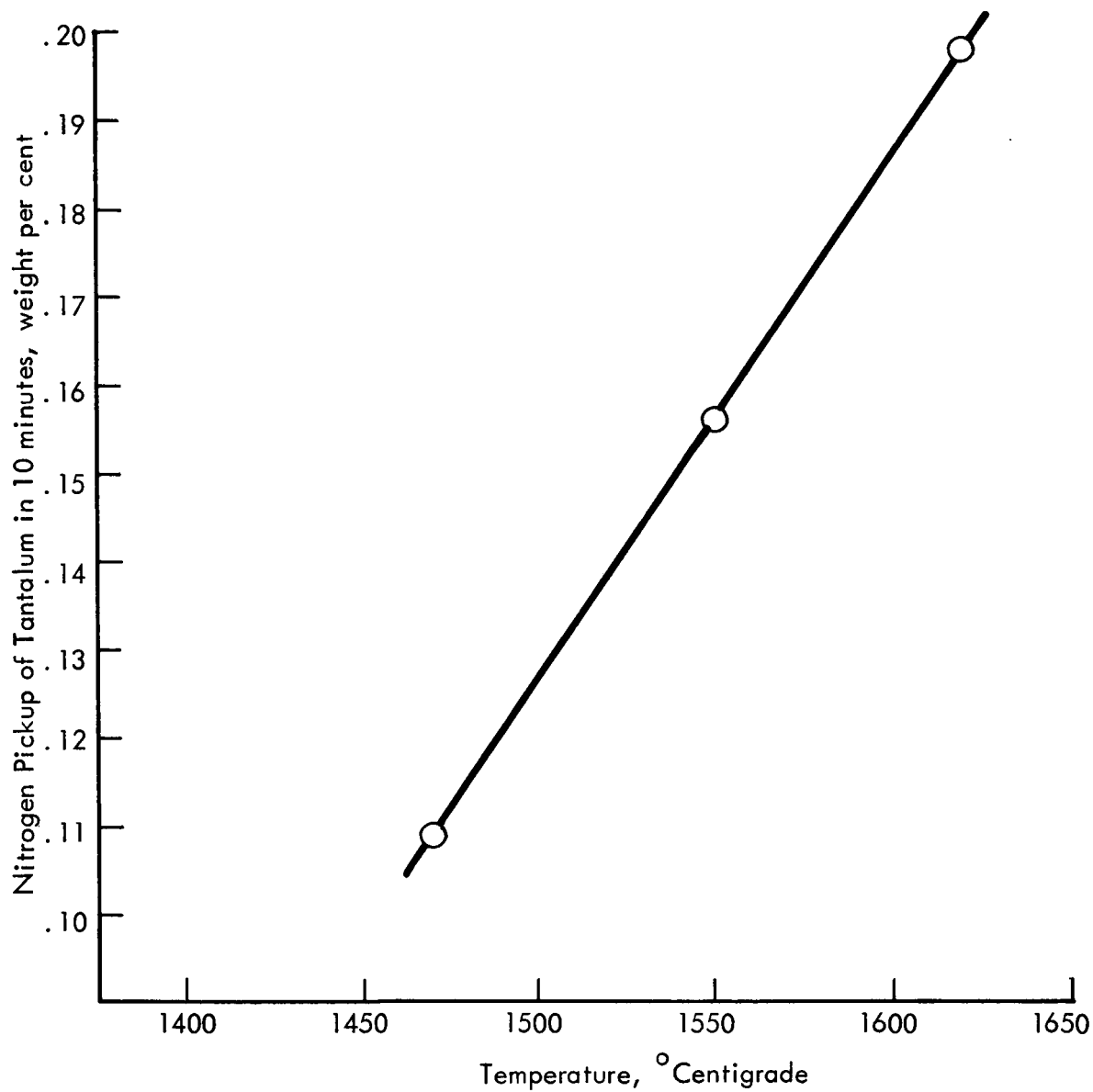


FIGURE 8 - Effect of Temperature on Nitrogen Pick-Up of Unalloyed Tantalum after 10 Minutes at Temperature and at  $p_{N_2} = 0.194$  Atmospheres.

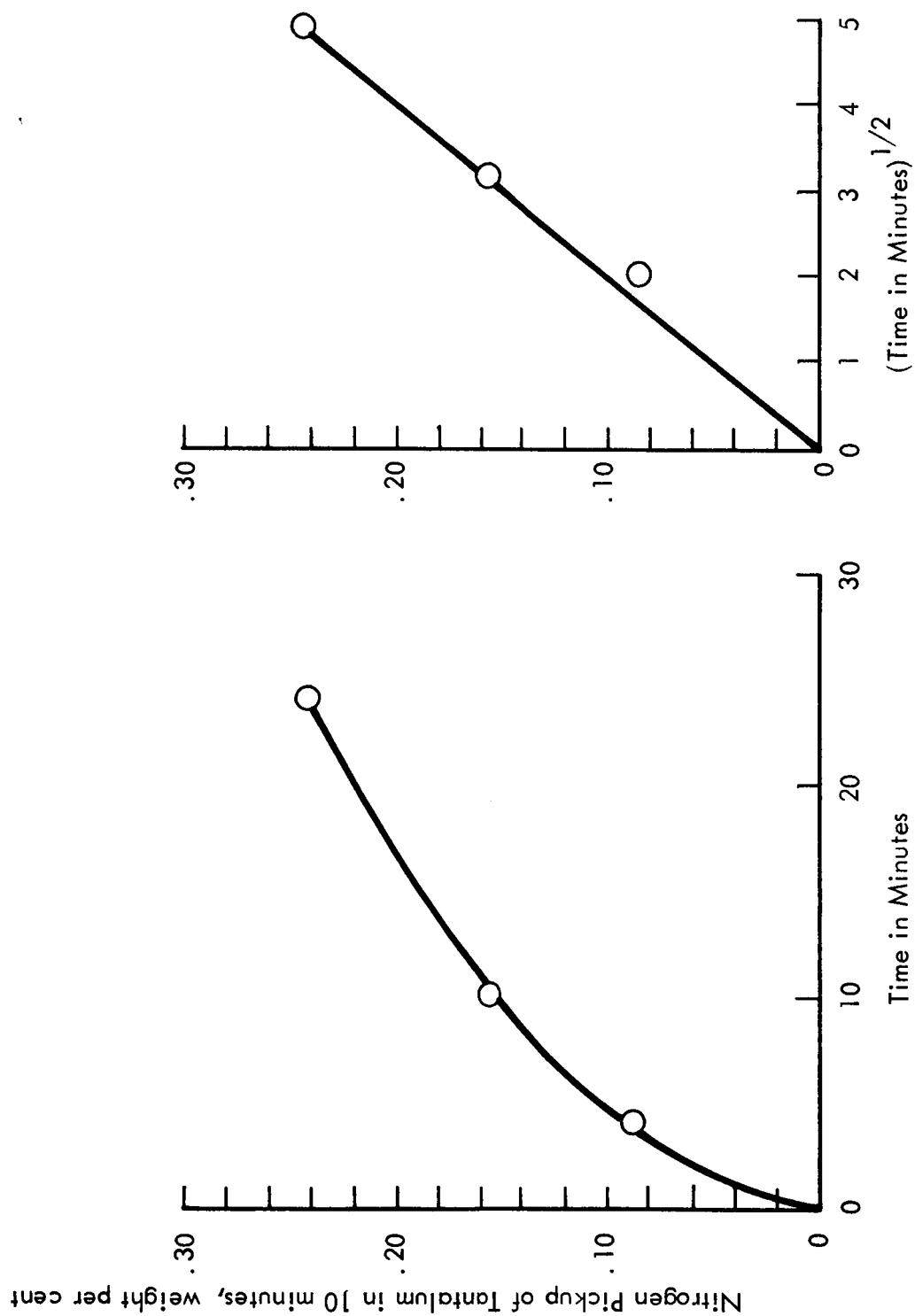


FIGURE 9 - Effect of Time on Nitrogen Pick-Up of Unalloyed Tantalum at 1550°C (2820°F) and at  $p_{N_2} = 0.194$  Atmospheres

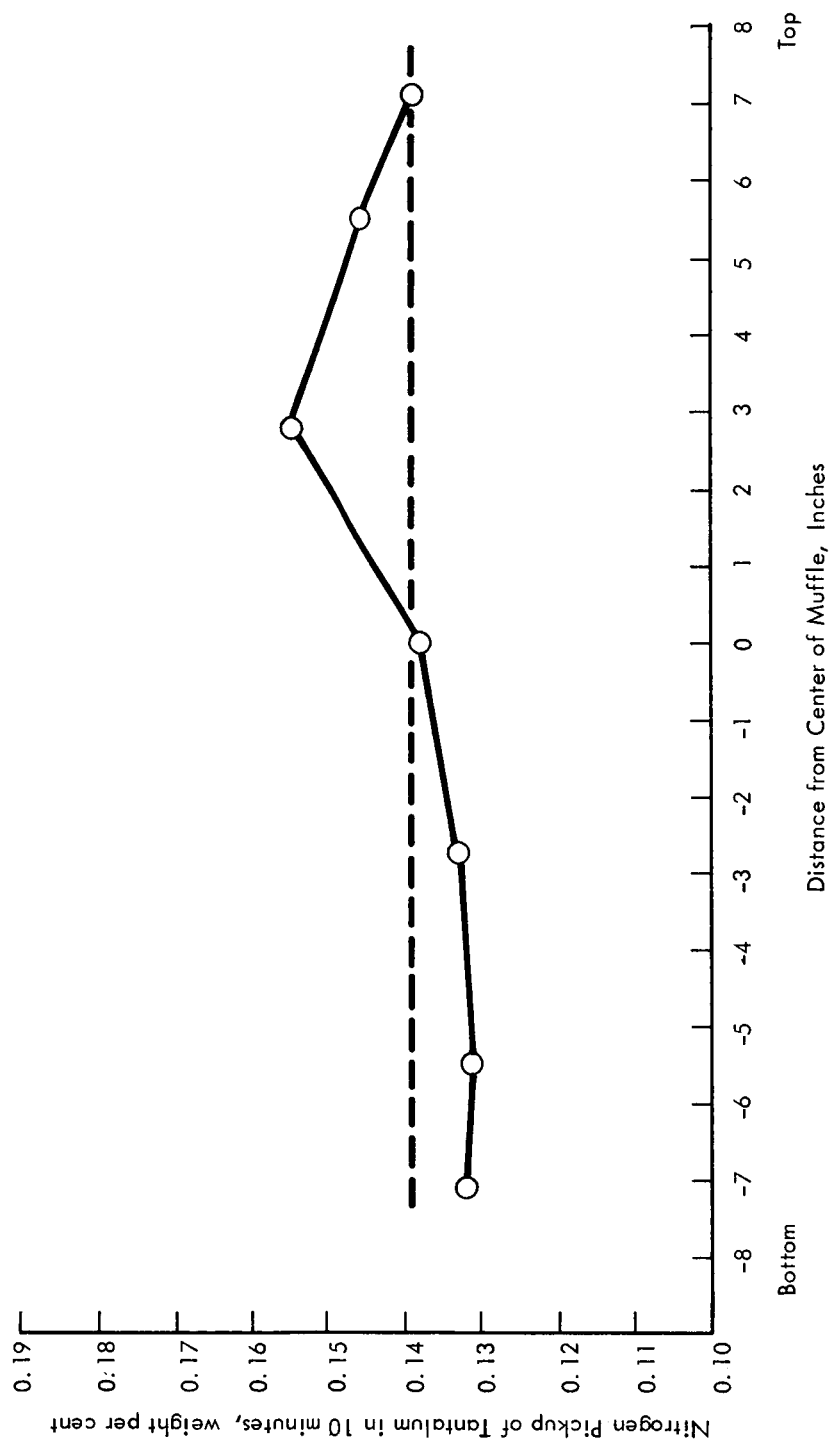


FIGURE 10 - Effect of Specimen Position in Furnace on Nitrogen Pick-Up of Unalloyed Tantalum (Dashed Line is Average Nitrogen Pick-Up for 7 Specimen Furnace Charge) at  $p_{N_2} = 0.194$  Atmospheres



the as-quenched condition, all five compositions were single phase when examined optically at 1500X. Specimens from each solution annealed composition were aged for one hour at temperatures from 700-1600°C (1290-2910°F). Changes in microstructure are being observed by both optical and electron microscopy, and Vickers indentation is being used to determine room temperature hardness changes. The change in room temperature hardness of the solution annealed material after one hour aging treatments is shown in Figure 11. Significant increases in the hardness of NAS-42 and 49 were measured without observing any microstructural changes. Precipitates were first observed in the grain boundaries after one hour at 1300°C (2370°F). General precipitation throughout the grain volume was observed after one hour at 1400°C (2550°F). The appearance of the general precipitate coincided with the observed hardness minima. The precipitating phase is extremely fine and was very difficult to resolve optically at 1500X. Figure 12 is a series of photomicrographs which show the sequence of precipitation. The hardness results and metallographic observation indicate that both NAS-42 and 49 follow classical precipitation hardening behavior. Examination by electron microscopy and x-ray diffraction is currently underway. The precipitating phase is probably ZrN, which is coherent with the matrix up to approximately 1300°C (2370°F). The behavior observed for the Ta-W-Zr-N and Ta-W-Mo-Re-Zr-N alloys is similar to that observed for the Cb-Hf-N system by Begley, et al.<sup>4</sup> The hardness level in the overaged condition is probably attributable to the high inherent nitrogen solubility in the tantalum alloy matrix. The composition NAS-39, which contains 300 ppm nitrogen, was single phase after heating the solution annealed material for one hour at all aging temperatures investigated. Figure 13 contains photomicrographs of NAS-39 after various aging treatments. Lack of any significant change in room temperature hardness and retention of the single phase microstructure in evidence of the relatively high nitrogen solubility in the tantalum alloy matrix. Seghezzi<sup>5</sup> reported a nitrogen solubility of 7 atom percent (0.58 w/o) in pure tantalum at 1600-2000°C (2910-3630°F) and Bauer and Zapp<sup>6</sup> report a nitrogen solubility in pure tantalum of 0.11 and 0.44 weight percent at 600°C (1110°F) and 1200°C (2190°F) respectively.

NAS-21, in the solution annealed condition, was single phase and had a hardness of 255 DPH. However, heating for one hour at 700°C (1219°F) resulted in a significant decrease in the room temperature hardness. A minima in room temperature hardness occurred after heating for one hour at 1000-1300°C (1830-2370°F). The slight hardness peak observed after the 1400°C (2550°F) treatment is not readily explained since metallographic observation showed coarsening of the dispersed carbide phase (See Figure 14).

Compositions NAS-36 and NAS-44 have nitrogen substituted for part of the carbon. Neither composition showed any significant changes in room temperature hardness over the range of aging temperatures investigated. Precipitation was observed in both compositions after aging at 800°C (1470°F) and the precipitate is presumed to be a carbide.

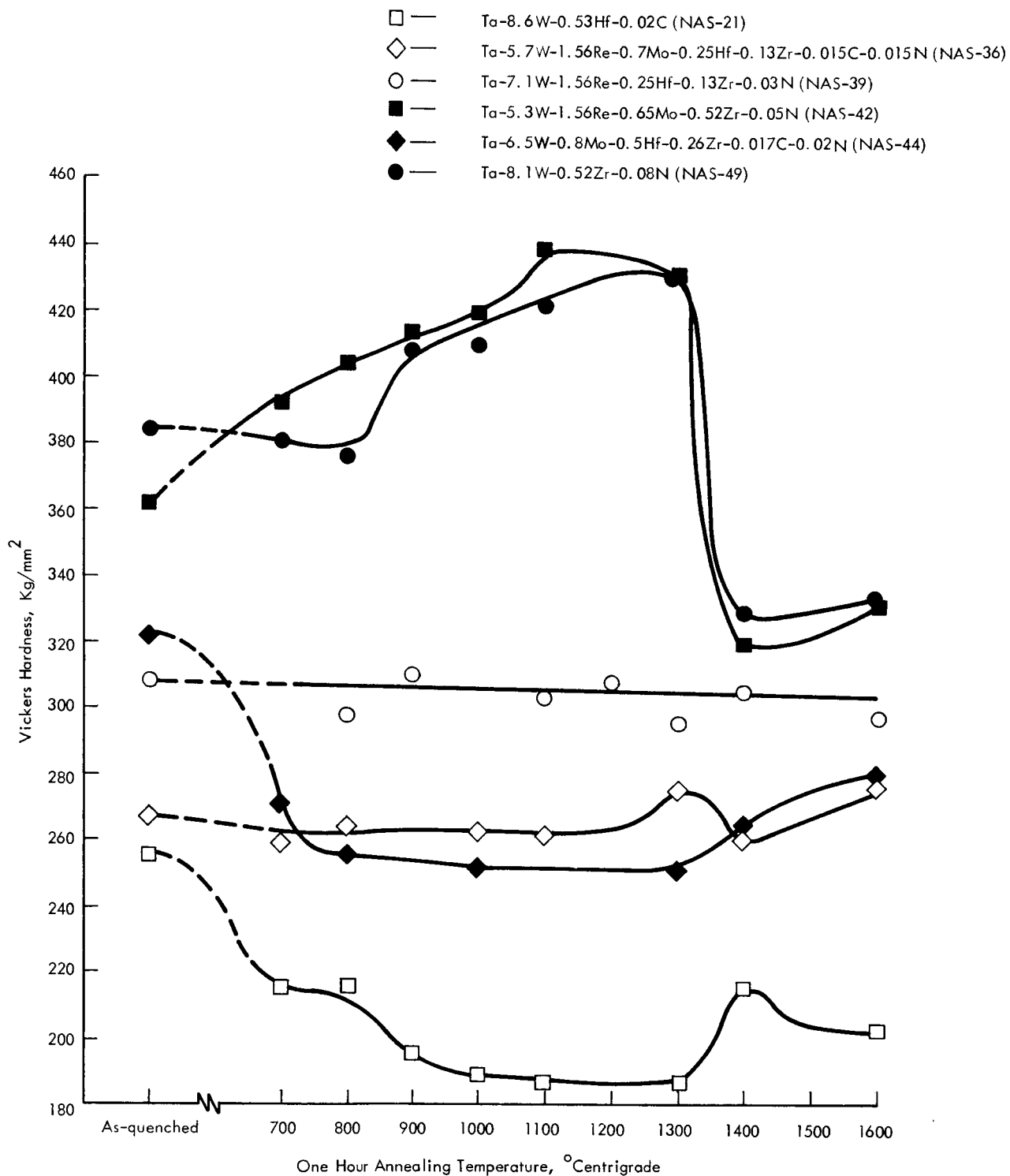
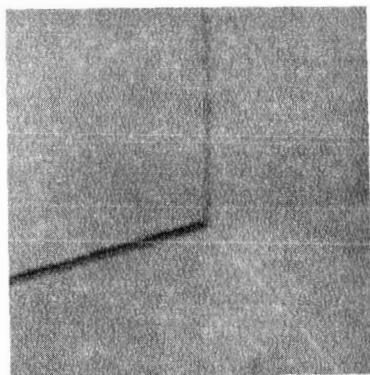
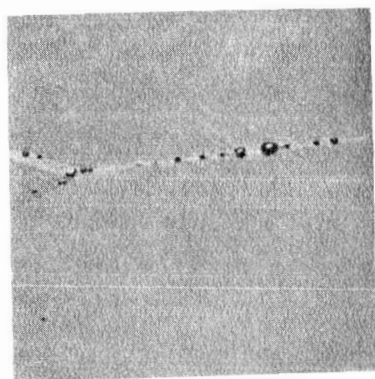


FIGURE 11 - Response of Tantalum Base Alloys to Heat Treatment



(a) Aged One Hour at 1100°C (2000°F)

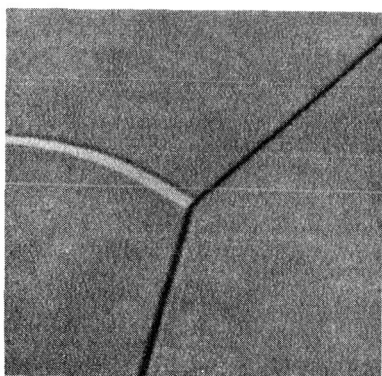


(b) Aged One Hour at 1300°C (2370°F)

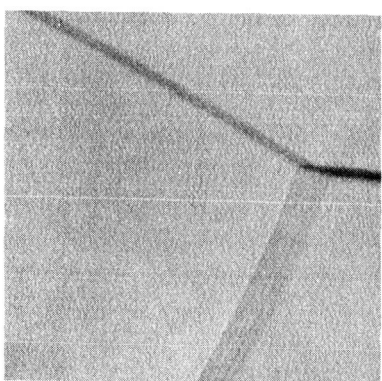


(c) Aged One Hour at 1400°C (2550°F)

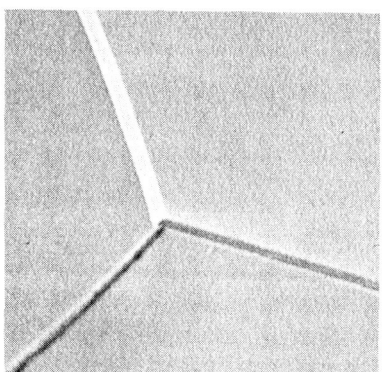
FIGURE 12 - Microstructure of NAS-42 (Ta-5.3W-1.56Re-0.65Mo-0.52Zr-0.05N)  
After Solution Annealing and Aging Etchant -  $\text{NH}_4\text{HF}-\text{HF}-\text{HNO}_3-\text{H}_2\text{O}$   
Mag. 1500X



(a) Annealed 1 Hour at 2000°C (3630°F)  
He Quenched



(b) Plus 1 Hour at 1100°C (2000°F)



(c) Plus 1 Hour at 1300°C (2370°F)

FIGURE 13 - Microstructure of NAS-39 (Ta-7.1W-1.56Re  
-0.25Hf-0.13Zr-0.03N) Etchant -  $\text{NH}_4\text{F} \cdot \text{HF}$ -  
 $\text{HNO}_3\text{-H}_2\text{O}$  Mag. 1500X

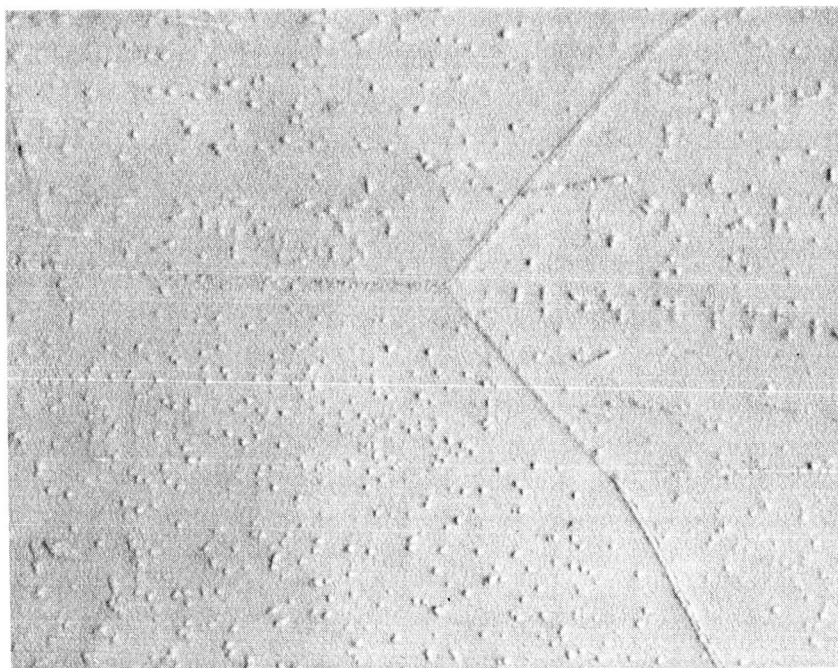


FIGURE 14 - Microstructure of NAS-21 (Ta-8.6W-0.53Hf-0.02C) After 1 Hour at 2000°C (3630°F), Helium Quenched Plus 1 Hour at 1400°C (2550°F). Etchant  $\text{NH}_4\text{F} \cdot \text{HF} - \text{HNO}_3 - \text{H}_2\text{O}$  Mag. 1500X

Investigation of the dispersed phase stability, particularly of the nitrogen bearing alloys, was continued on compositions NAS-36, 39, and 42. Specimens of each in the various initial conditions described in Table VI were aged for 500 hours at 1150°C (2100°F) and 1250°C (2280°F). Samples of the solution treated material were given reductions of 2-1/2 and 5 percent to increase dislocation density, thereby providing additional nucleation sites. The aged specimens are being examined using optical and electron metallographic techniques. Precipitated phases will be extracted chemically and identified by x-ray diffraction.

Varian ultra-high vacuum annealing furnaces were used to retard oxygen contamination of the tantalum alloy specimens during the extended aging treatments. A complete description of the furnace and operation is described elsewhere.<sup>7</sup> Samples of each composition were etched, rinsed, dried, and wrapped in tantalum foil prior to loading into the furnace. After evacuation of the furnace chamber and system bakeout at 250°C (480°F) for 31 hours, the furnace and load was brought to temperature. Four hours after reaching the desired test temperature, the system pressure was less than  $5 \times 10^{-9}$  torr, and at the termination of the 500 hour test, the pressure was less than  $5 \times 10^{-10}$  torr. Temperature was monitored with a Pyro micro-optical pyrometer sighting into a black body cavity attached to the specimen.

TABLE VI - Vickers Hardness (a) in the Initial Condition of Tantalum Alloys Prior to 500 Hour Aging Treatment

Condition	Composition (Heat Number)		
	Ta-5.7W-1.56Re-0.7Nb -0.25Hf-0.13Zr-0.015N -0.015C (NAS-36)	Ta-7.1W-1.56Re-0.25Hf -0.13Zr-0.03N (NAS-39)	Ta-5.3W-1.56Re-0.65Mo -0.52Zr-0.05N (NAS-42)
As-Worked, 33% Prior Reduction	358	---	458
As-Annealed 1 Hr. at 1650°C (3000°F)	297	330	---
As-Annealed 1 Hr. at 1650°C (3000°F) + 2-1/2% Red.	323	389	---
As-Annealed 1 Hr. at 1650°C (3000°F) + 5% Red.	338	363	---
As-Annealed 1 Hr. at 2000°C (3630°F)	327	326	368
As-Annealed 1 Hr. at 2000°C (3630°F) + 2-1/2% Red.	388	354	425
As-Annealed 1 Hr. at 2000°C (3630°F)	362	379	438
As-Forged	---	---	409
As-Forged + 1 Hr. at 2000°C (3630°F)	---	---	---

(a) 30 Kg. Load

### III. FUTURE WORK

During the next quarterly period it is planned to accomplish the following:

1. Evaluation of the 2 inch diameter ingot forging composition will be completed.
2. Compositions for the optimization investigation will be selected and melting of 2 inch diameter ingots initiated.
3. Phase identification and response to thermal treatment investigations will continue.



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